

Determination of Potassium in Tobacco

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A method for potassium in tobacco, based on extraction with 0.5*N* HCl and flame photometry of the extract, was evaluated by a collaborative test between 12 laboratories. Results indicate that more work on the method is needed. The coefficients of variation for the six samples increased as the per cent potassium increased. The distribution of values for the separate samples indicates that the flame photometry rather than the extraction procedure was responsible for the lack of good precision between laboratories on the samples high in potassium. Modifications are suggested for improving the method.

A simple, rapid procedure for potassium in tobacco, developed by Dobbins (*This Journal*, 44, 360 (1961)), was subjected to collaborative testing. The method is based on the column extraction of the potassium ion in 0.5*N* HCl, followed by flame photometric measurement on the column eluate.

Twelve collaborators analyzed six samples in duplicate and reported all data. The samples were Burley and Flue-cured stems, and Burley, Flue-cured, Maryland, and Wisconsin leaf.

METHOD

Reagents

(a) *Potassium stock solution*.—2,000 ppm. Dissolve 3.8134 g ACS grade KCl in water and dilute to 1 L. Store in polyethylene bottle.

(b) *Potassium standard solutions*.—Prepare 0, 10, 20, and 30 ppm K solutions as follows, and store in polyethylene bottles: (1) 0 ppm—Dilute 20 ml 0.5*N* HCl to 1 L; (2) 10 ppm—Dilute 5 ml K stock soln and 20 ml 0.5*N* HCl to 1 L; (3) 20 ppm—Dilute 10 ml K stock soln and 20 ml 0.5*N* HCl to 1 L; (4) 30 ppm

—Dilute 15 ml K stock soln and 20 ml 0.5*N* HCl to 1 L.

Apparatus

(a) *Flame photometer*.—Perkin-Elmer model 146¹ or equivalent, preferably with natural gas-air flame and an atomizer with 10 ml/min. flow.

(b) *Chromatographic column*.—20 × 150 mm with coarse frit.

Determination

Accurately weigh about 0.25 g tobacco ground to pass a 40-mesh sieve into weighing pan of about 40 ml capacity. Add approximately 1 g untreated Celite 545¹ and intimately mix with spatula. Transfer through powder funnel into chromatographic tube, insuring quantitative transfer by brushing or tapping weighing pan and funnel. Add additional Celite to tube until 1" layer accumulates on top of sample-Celite mixture. Compact sample and Celite by briskly tapping tip of tube against wooden table top and insert tip of the tube into 1 L volumetric flask. Pipet 20 ml 0.5*N* HCl into tube, washing down sides of tube during addition, and allow to elute through sample-Celite mixture into volumetric flask. When the liquid level reaches top of Celite, similarly add 25 ml water and allow to elute through. Add second 25 ml portion of water to tube and allow to elute normally, or force through rapidly with compressed air. Rinse tip of chromatographic tube into volumetric flask, and dilute to volume with water.

Determine % *T* for unknown eluate and potassium standards by procedures specified in instruction manual of flame photometer used.

Prepare calibration curve from 0 to 30 ppm potassium standards by plotting % *T* vs. ppm potassium. Read % *T* of unknown sample, and determine ppm K from calibration curve.

¹ Mention of a specific company's product does not imply endorsement of this product by the Department of Agriculture over any other similar products not named.

Table 1. Collaborative results on the determination of potassium in tobacco

Coll. No.	Sample Number ^a						Std Dev., %
	1	2	3	4	5	6	
1	11.40	6.88	5.75	3.75	5.28	6.59	0.06
4	11.95	6.94	5.78	3.66	5.40	6.75	0.00
5	13.07	7.42	5.56	4.16	6.02	7.13	0.06
9	9.95	7.02	5.76	3.77	5.30	6.65	0.27
11	11.47	7.04	5.70	3.65	5.25	6.68	0.06
12	11.43	6.43	5.24	3.20	4.70	6.41	0.06
13	11.64	6.98	5.59	3.66	5.32	6.66	0.01
14	11.55	7.02	5.51	3.63	5.33	6.76	0.04
15	12.70	7.65	5.48	3.73	5.12	6.06	0.02
22	11.09	6.99	5.54	3.74	5.54	6.99	0.07
23	10.79	6.72	5.72	3.78	5.31	6.56	0.13
24	12.43	6.58	6.27	3.85	6.02	(8.18)	0.02
\bar{X}	11.61	7.01	5.66	3.72	5.38	6.66	
<i>s</i>	0.88	0.33	0.25	0.22	0.36	0.28	
\bar{X}^b	11.37	7.03	5.65	3.71	5.32	6.63	
<i>s</i> ^b	0.76	0.25	0.12	0.06	0.11	0.25	
C. V. % ^b	6.7	3.6	2.1	1.6	2.1	3.8	

^a 1, Burley stems; 2, Flue-cured stems; 3, Burley leaf; 4, Flue-cured leaf; 5, Maryland leaf; 6, Wisconsin leaf.
^b Data from Collaborators 5, 12, and 24 were eliminated.

Calculate % K = ppm K \times 0.1/sample wt (g).

% K₂O = ppm K \times 0.1205/sample wt (g).

Results

Twelve collaborators analyzed the six samples in duplicate and reported all data. The mean of the duplicate values, over-all sample means, and within- and between-laboratory standard deviations are shown in Table 1. Three of the collaborators were either consistently low or high; this deviation indicated some inherent error in their analyses. The interlaboratory precisions were calculated again, but with these three sets of data omitted; results are shown in Table 1. This led to markedly improved precision for three of the six samples and a small improvement in the other three.

The coefficients of variation in per cent increased systematically as the per cent potassium increased from 1.6 for the sample with 3.72% K to 6.7 at the 11.37% K level. A systematic error in the method would be expected to produce coefficients of variation

which either diminished with increase in potassium level or were relatively constant. The poor precision on the samples high in potassium was caused by several values being either much higher or much lower than the majority. Apparently incomplete extraction must be eliminated as the cause of the lack of agreement between laboratories; flame photometry is the probable source of error. It may be that there were both positive and negative interferences with solutions containing higher salt concentrations, depending on the instrument and flame used, or that the instrument lacked stability at the higher concentrations even though all sample solutions fell within the calibration range. It would seem advisable to repeat the study of this method, and this time to limit the range of concentration from 0 to 15 ppm K and to modify the directions to include frequent checking of the calibration curve. Because the flame intensity or sample aspiration rate may vary, it is often advisable to introduce a standard sample at fre-

quent intervals during a series of analyses and correct the sample readings for any shift in the % transmission for the standard.

The collaborators were asked to express their preference as to whether the results should be reported as % K or K_2O . Seven preferred % K and five chose % K_2O , so the calculations for both were included in the method.

It is recommended that the study of potassium in tobacco be continued.

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